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Annual Progress Report for
Advanced Processing of Hollow Sphere Foams

June 1, 1998 to May 31, 1999

by

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Advanced Processing of Hollow Sphere Foams and the Development of Alternate Processing Schemes for Ultra-Low Density Steel and Titanium Alloys

August 6, 1999

Executive Summary

Closed-cell metallic foams were fabricated by bonding millimeter sized hollow spheres at points of contact. The composition of low density metallic foams with hollow sphere architecture achieved to date includes Ti-6Al-4V, 400 series stainless steel, and Inconel 718. Hollow spheres are formed as powder shells from slurries of non-metallic precursor powders at room temperature. Subsequent heat treatment coupled with either direct reduction or hydride-dehydride processing converts precursors into the appropriate metal alloys. To produce titanium alloy spheres, the starting powder is titanium alloy hydride. Thermal treatment in an inert atmosphere decomposes the hydride and sinters the titanium powder in the sphere walls to greater than 96% relative density. Both titanium and Ti-6V-4V spheres and foams have been produced. However, oxygen content remains a concern for the titanium compositions explored and the titanium alloy foams produced so far behave like brittle foam. For stainless steel spheres, the starting powder is a mixture of iron and chromium oxide. Thermal treatment in hydrogen reduces the oxides to Fe-Cr alloys with less than 2% porosity in sphere walls. The nominal composition is close to that of 405 stainless. Carburization in CO/CO₂ atmosphere followed by heat treatment produces foams of either 410 or 420 type stainless steels depending on carbon content. Compressive stress-strain behavior was measured on point contact bonded stainless foams both before and after carburization. A sample with ~0.5 wt% carbon at a relative density of 15% indicated a yield strength of 16 MPa. Specific strengths of the foams were positioned between open and closed cell models. This was encouraging because bonding in the foams was less than optimum and the hollow sphere walls contained numerous defects. With improvements in processing, strengths should increase while maintaining ductility. Several other steel compositions are being investigated as to their viability to undergo the direct reduction process as well. These include: a maraging steel, an austenitic stainless steel, a duplex stainless steel, and a precipitation-hardened stainless steel. Inconel 718 with a composition of Ni 19Cr 18Fe 5.1Nb 3Mo 0.9Al 0.6Ti 0.05C was selected to demonstrate the compositional versatility of this unique fabrication process. Inconel foams were processed in similar fashion as the stainless steels. Hollow spheres and foams from Inconel 718 with bulk density ranging from 0.6 to 1.1 g/cc and 1.0 to 2.1 g/cc, respectively, were fabricated.

There were two major concerns that emerged out of this research. First, to fabricate a foam with adequate ductility in either commercially pure titanium or Ti6Al-4V, the steps in the process where oxygen is being introduced must be identified, controls at these points must be made to reduce the possibility of oxygen contamination. As an alternative, titanium alloys which are not as sensitive to oxygen contamination should be considered. For example, high β alloys might be lightly candidates.

A second issue that must be addressed and bears on all alloys fabricated using the room temperature slurry process is sphere wall uniformity and defect content. Currently, the uniformity in thickness of the steel foams is of particular concern and we anticipate that the solution of this problem will be difficult especially in the systems fabricated from mixtures of oxides of iron and chromium. In the next program, alternatives to the hollow sphere process will be explored to produce low density structures with more sphere wall uniformity and lower defect density.

Manuscripts are attached for the recent publications supported by this program which are listed as follows:

Publications:

1. C. Uslu, K.J. Lee, T.H. Sanders, J. K. Cochran, "Ti-6Al-4V Hollow Sphere Foams", Synthesis/Processing of Light Weight Metallic Materials II, Edited by Malcolm Ward-Close, TMS, Warrendale, PA, pp 289-300, September, 1997.
2. A.R. Nagel, C. Uslu, K.J. Lee, J. K. Cochran, T. H. Sanders, "Steel Closed Cell Foams from Direct Oxide Reduction", Synthesis/Processing of Light Weight Metallic Materials II, Edited by Malcolm Ward-Close, TMS, Warrendale, PA, pp 395-406, September, 1997.
3. N. Baxter, C. Uslu, K.J. Lee, T.H. Sanders, J. K. Cochran, "Hollow Sphere Metal Foams", Synthesis/Processing of Light Weight Metallic Materials II, Edited by Malcolm Ward-Close, TMS, Warrendale, PA, pp 417-430, September, 1997.
4. C. U. Hardwicke, J. K. Cochran, and T. H. Sanders, "Rheological Properties of Concentrated, Nonaqueous Titanium Hydride Suspensions", *Colloids and Surfaces A: Physicochemical and Engineering Aspects*, final revisions completed.
5. K. M. Hurysz, J. L. Clark, A.R. Nagle, C. U. Hardwicke, K.J. Lee, J. K. Cochran, and T.H. Sanders, Jr. "Steel and Titanium Hollow Sphere Foams", Porous and Cellular Materials for Structural Applications, A. Evans, D. Schwartz, D. Shih, and H. Wadley, eds., MRS Proceedings, V-521, pp 191-203, Pittsburg, PA, April, 1998.
6. Joe K. Cochran, "Ceramic Hollow Spheres and Their Applications", *Current Opinion in Solid State & Materials Science*, vol 3, no 5, pp 474-479, Oct. 1998. (Invited Article)
7. K. M. Hurysz, J. K. Cochran, K. J. Lee, T. H. Sanders, J. L. Clark, "Dual Adsorption on Negatively Charged Surfaces in Non-Aqueous Media", *Symposium on Innovative Processing and Synthesis of Ceramics, Glasses and Composites*, American Ceramic Society, Indianapolis, IN, April, 1999, inpress.
8. J. L. Clark, J. K. Cochran, K J Lee, T. H. Sanders, "Stainless Steel Hollow Sphere Foams - Fabrication, Carburization and Properties", *Proceedings MetFoam 99*, Bremen Germany, June 13-15, 1999, 8 pages.

9. J. Nadler, J. L. Clark, J. K. Cochran, K. J. Lee, T. H. Sanders, "Fabrication and Microstructure of Metal-metal Syntactic Foams", Proceedings MetFoam 99, Bremen Germany, June 13-15, 1999.

10. T. H. Sanders, Jr., J. K. Cochran, K. J. Lee, J. L. Clark, K. M. Hurysz, and Wes Seay "Unique Methods of Fabricating Ultra-Low Density Steel Microstructures", Professor George Krauss International Symposium, ASM Annual Meeting, 1-4 November, Cincinnati, Ohio, in preparation.

Theses:

11. Canan U. Hardwicke, "Processing and Properties of Ti-6Al-4V Hollow Sphere Foams from Hydride Powders", Ph.D. Dissertation, Materials Science and Engineering, Georgia Institute of Technology, September, 1997.

12. Adam R. Nagel, "Closed Cell Steel Foams from Oxide Reduction", MS Thesis, Materials Science and Engineering, Georgia Institute of Technology, December, 1997.

13. Kevin M. Hurysz, "Titanium Hollow Sphere Foams of Improved Quality", MS Thesis, Materials Science and Engineering, Georgia Institute of Technology, September, 1998.

14. Justin L. Clark, "Stainless Steel Hollow Sphere Foams- Processing, Properties and Microstructure", MS Thesis, Materials Science and Engineering, Georgia Institute of Technology, in preparation.